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W05078 I <sup>Dayes</sup> 4/26/12

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Response to resubmittal  
request.  
Rec  
3/19/07  
DP

Analytical Data Package Prepared For  
**Pacific Northwest National Lab**

Radiochemical Analysis By

**STL Richland STLRL**

*2800 G.W. Way, Richland Wa, 99354, (509)-375-3131.*

*Data Package Contains \_\_\_\_\_ Pages*

**Report Nbr: 34589**

SDG Nbr	ORDER Nbr	CLIENT ID NUMBER	LOT Nbr	WORK ORDER	RPT DB ID	BATCH
W05078 I	S07-010	BIKPD6	J6L180167-1	JLPEJ1AA	9JLPEJ10	6353652

*Dayes*  
*4/26/12*

---

Comments:



STL

STL Richland  
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Richland, WA 99354

Tel: 509 375 3131 Fax: 509 375 5590  
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## Certificate of Analysis

Pacific Northwest National Laboratories  
Sigma V Building  
Richland, WA 99352

February 28, 2007

Attention: Dot Stewart

---

SAF Number	:	S07-010
Date SDG Closed	:	December 14, 2006
Number of Samples	:	One (1)
Sample Type	:	Water
SDG Number	:	W05078
Data Deliverable	:	45-Day / Summary

---

### CASE NARRATIVE

#### I. Introduction

On December 14, 2006, one water sample was received at STL Richland (STLR) for radiochemical analysis. Upon receipt, the sample was assigned the following laboratory ID number to correspond with the Pacific Northwest National Laboratories (PGW) specific ID:

<u>PGW ID#</u>	<u>STLR ID#</u>	<u>DATE OF RECEIPT</u>	<u>MATRIX</u>
B1KPD6	JLPEJ	12/14/06	WATER

#### II. Sample Receipt

The samples were received in good condition and no anomalies were noted during check-in.

#### III. Analytical Results/Methodology

The analytical results for this report are presented by laboratory sample ID. Each set of data includes sample identification information, analytical results and the appropriate associated statistical errors.

The requested analyses were:

**Liquid Scintillation Counting**  
Enriched Tritium by method RICH-RC-5024

**IV. Quality Control**

The analytical results for each analysis performed includes a minimum of one laboratory control sample (LCS), one method (reagent) blank, and one duplicate sample analysis. Any exceptions have been noted in the "Comments" section.

QC and sample results are reported in the same units.

**V. Comments**

**Liquid Scintillation Counting**

Enriched Tritium by method RICH-RC-5024

The LCS, batch blank, sample and sample duplicate are all within contractual limits.

I certify that this Certificate of Analysis is in compliance with the SOW, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hard copy data package has been authorized by the Laboratory Manager, or a designee as verified by the following signature.

Reviewed and approved:



Sherryl A. Adam  
Project Manager

## Drinking Water Method Cross References

DRINKING WATER ASTM METHOD CROSS REFERENCES		
Referenced Method	Isotope(s)	STL Richland's SOP number
EPA 901.1	Cs-134, I-131	RICH-RC-5017
EPA 900.0	Alpha & Beta	RICH-RC-5014
EPA 903.1	Ra-226	RICH-RC-5005
EPA 904.0	Ra-228	RICH-RC-5005
EPA 905.0	Sr89/90	RICH-RC-5006
ASTM D2460	Total Radium	RICH-RC-5027
Standard Method 7500-U-C & ASTM D5174	Uranium	RICH-RC-5058
EPA 906.0	Tritium	RICH-RC-5007
NOTE:		
The Gross Alpha LCS is prepared with Am-241 (unless otherwise specified in the case narrative)		
The Gross Beta LCS is prepared with Sr/Y-90 (unless otherwise specified in the case narrative)		

## Uncertainty Estimation

STL Richland has adopted the internationally accepted approach to estimating uncertainties described in "NIST Technical Note 1297, 1994 Edition". The approach, "Law of Propagation of Errors", involves the identification of all variables in an analytical method which are used to derive a result. These variables are related to the analytical result (R) by some functional relationship,  $R = \text{constants} * f(x, y, z, \dots)$ . The components (x, y, z) are evaluated to determine their contribution to the overall method uncertainty. The individual component uncertainties ( $u_i$ ) are then combined using a statistical model that provides the most probable overall uncertainty value. All component uncertainties are categorized as type A, evaluated by statistical methods, or type B, evaluated by other means. Uncertainties not included in the components, such as sample homogeneity, are combined with the component uncertainty as the square root of the sum-of-the-squares of the individual uncertainties. The uncertainty associated with the derived result is the combined uncertainty ( $u_c$ ) multiplied by the coverage factor (1, 2, or 3).

When three or more sample replicates are used to derive the analytical result, the type A uncertainty is the standard deviation of the mean value ( $S/\sqrt{n}$ ), where S is the standard deviation of the derived results. The type B uncertainties are all other random or non-random components that are not included in the standard deviation.

The derivation of the general "Law of Propagation of Errors" equations and specific example are available on request.

## Report Definitions

<b>Action Lev</b>	An agreed upon activity level used to trigger some action when the final result is greater than or equal to the Action Level. Often the Action Level is related to the Decision Limit.
<b>Batch</b>	The QC preparation batch number that relates laboratory samples to QC samples that were prepared and analyzed together.
<b>Bias</b>	Defined by the equation (Result/Expected)-1 as defined by ANSI N13.30.
<b>COC No</b>	Chain of Custody Number assigned by the Client or STL Richland.
<b>Count Error (#s)</b>	Poisson counting statistics of the gross sample count and background. The uncertainty is absolute and in the same units as the result. For Liquid Scintillation Counting (LSC) the batch blank count is the background.
<b>Total Uncert (#s) <i>u<sub>c</sub> - Combined Uncertainty.</i></b>	All known uncertainties associated with the preparation and analysis of the sample are propagated to give a measure of the uncertainty associated with the result, <i>u<sub>c</sub> the combined uncertainty</i> . The uncertainty is absolute and in the same units as the result.
<b>(#s), Coverage Factor</b>	The coverage factor defines the width of the confidence interval, 1, 2 or 3 standard deviations.
<b>CRDL (RL)</b>	Contractual Required Detection Limit as defined in the Client's Statement Of Work or STL Richland "default" nominal detection limit. Often referred to the reporting level (RL)
<b>Lc</b>	Decision Level based on instrument background or blank, adjusted by the Efficiency, Chemical Yield, and Volume associated with the sample. The Type I error probability is approximately 5%. $Lc = (1.645 * \sqrt{2 * (BkgndCnt / BkgndCntMin) / SCntMin}) * (ConvFct / (Eff * Yld * Abn * Vol)) * IngrFct$ . For LSC methods the batch blank is used as a measure of the background variability. Lc cannot be calculated when the background count is zero.
<b>Lot-Sample No</b>	The number assigned by the LIMS software to track samples received on the same day for a given client. The sample number is a sequential number assigned to each sample in the Lot.
<b>MDC MDA</b>	Detection Level based on instrument background or blank, adjusted by the Efficiency, Chemical Yield, and Volume with a Type I and II error probability of approximately 5%. $MDC = (4.65 * \sqrt{((BkgndCnt / BkgndCntMin) / SCntMin) + 2.71 / SCntMin}) * (ConvFct / (Eff * Yld * Abn * Vol)) * IngrFct$ . For LSC methods the batch blank is used as a measure of the background variability.
<b>Primary Detector</b>	The instrument identifier associated with the analysis of the sample aliquot.
<b>Ratio U-234/U-238</b>	The U-234 result divided by the U-238 result. The U-234/U-238 ratio for natural uranium in NIST SRM 4321C is 1.038.
<b>Rst/MDC</b>	Ratio of the Result to the MDC. A value greater than 1 may indicate activity above background at a high level of confidence. Caution should be used when applying this factor and it should be used in concert with the qualifiers associated with the result.
<b>Rst/TotUcert</b>	Ratio of the Result to the Total Uncertainty. If the uncertainty has a coverage factor of 2 a value greater than 1 may indicate activity above background at approximately the 95% level of confidence assuming a two-sided confidence interval. Caution should be used when applying this factor and it should be used in concert with the qualifiers associated with the result.
<b>Report DB No</b>	Sample Identifier used by the report system. The number is based upon the first five digits of the Work Order Number.
<b>RER</b>	The equation Replicate Error Ratio = $(S - D) / [\sqrt{(TPUs^2 + TPUD^2)}]$ as defined by ICPT BOA where S is the original sample result, D is the result of the duplicate, TPUs is the total uncertainty of the original sample and TPUD is the total uncertainty of the duplicate sample.
<b>SDG</b>	Sample Delivery Group Number assigned by the Client or assigned by STL Richland upon sample receipt.
<b>Sum Rpt Alpha Spec Rst(s)</b>	The sum of the reported alpha spec results for tests derived from the same sample excluding duplicate result where the results are in the same units.
<b>Work Order</b>	The LIMS software assign test specific identifier.
<b>Yield</b>	The recovery of the tracer added to the sample such as Pu-242 used to trace a Pu-239/40 method.

3/5/2007 11:16:31 AM

## STL Richland Report

Lab Code: STLRL

FormNbr: R      FormatType: FEAD      Version: 05      Rpt Nbr: 34589      File Name: h:\Reportdb\edd\Fead\VRad\W05078.Edd, h:\Reportdb\edd\Fead\VRad\34589.Edd

Lab Sample Id:	Client Id:	Test User	Contract Nbr	SAF Nbr	Sdg Nbr:	QC Type:	Moisture/ Solids%:	Distilled Volume	Sample On Date:	Collection Date:				
9JLPEJ10	B1KPD6		MW6-SBB-A1	S07-010	W05078					12/14/2006 09:23				
Batch	Analyte	CAS#	Result	Unit	CntU 2S	TotU 2S	Qual	MDA	TrcYield	Method	Alq Size	Unit	Analy Date/Time	Act
6353652	H-3	10028-17-8	1.55E+02	pCi/L	1.0E+01	2.9E+01		5.50E+00	100.0	TRITIUM_ELECT_L	1.50E-01	L	02/28/2007 02:58	I

STL Richland

rptFeadRadSummaryEdd v3.48

U Qual - Analyzed for, but the result is less than the Mdc or gamma scan did not identify the nuclide.  
 J Qual - No U qualifier has been assigned and the result is below the Reporting Limit (CRDL).  
 B Qual- Analyte was found in the associated laboratory blank above the MDC.

1

Monday, March 05, 2007

## STL Richland QC Blank Report

Lab Code: STLRL

FormNbr: R

FormatType: FEAD

VersionNbr: 05

File Name: h:\Reportdb\edd\Fead\Rad\W05078.Edd, h:\Reportdb\edd\Fead\Rad\34589.Edd

Lab Sample Id: JLTJW1AB

Sdg/Rept Nbr: W05078

34589

Collection Date: 12/14/2006 09:23

Client Id: NA

Matrix: WATER

WATER

Sample On Date:

Moisture/Solids%\*:

QC Type: BLK

Received Date: 12/14/2006

SAF Nbr	Contract Nbr	Test User	Case Nbr	SAS Nbr	Suffix	Decant	Distilled Volume	File Id	FSuffix	RTyp					
	MW6-SBB-A19981								AC	H					
Batch # / Qc Type	Analyt/ CAS#	Result/ Orig Rst	Unit	Tot/Cnt Uncert 2S	Qu- al	MDC	Tracer Yield	Spk Conc/ %Rec	Analy Method	Aliq Size/	Date/Time Analyzed	RPD/ UCL	RER/ UCL	LCS LCL/UCL	R Typ
6353652	H-3	7.54E+00	pCi/L	6.5E+00		5.47E+00	100.0		TRITIUM_ELE	1.50E-01	02/28/2007				D
BLK	10028-17-8			4.7E+00						L	00:23				

STL Richland

rptFeadRadEdd v3.68

U Qual - Analyzed for, but the result is less than the Mdc or gamma scan did not identify the nuclide.

J Qual - No U qualifier has been assigned and the result is below the Reporting Limit (CRDL).

B Qual- Analyte was found in the associated laboratory blank above the MDC.

1



Monday, March 05, 2007

## STL Richland QC Control Sample Report

Lab Code: STLRL

FormNbr: R

FormatType: FEAD

VersionNbr: 05

File Name: h:\Reportdb\edd\Fead\VRad\W05078.Edd, h:\Reportdb\edd\Fead\VRad\34589.Edd

Lab Sample Id: JLTJW1CS

Sdg/Rept Nbr: W05078

34589

Collection Date: 12/14/2006 09:23

Client Id: NA

Matrix: WATER

WATER

Sample On Date:

Moisture/Solids%\*:

QC Type: BS

Received Date: 12/14/2006

SAF Nbr		Contract Nbr		Test User		Case Nbr		SAS Nbr		Suffix		Decant		Distilled Volume		File Id		FSuffix		RTyp	
		MW6-SBB-A19981																AD		H	
Batch # / Qc Type	Analyt/ CAS#	Result/ Orig Rst	Unit	Tot/Cnt Uncert 2S	Qu- al	MDC	Tracer Yield	Spk Conc/ %Rec	Analy Method	Aliq Size/	Date/Time Analyzed	RPD/ UCL	RER/ UCL	LCS LCL/UCL	R Typ						
6353652	H-3	4.59E+02	pCi/L	8.1E+01		5.48E+00	100.0	4.49E+02	TRITIUM_ELE	1.5003E-01	02/28/2007			70	D						
BS	10028-17-8			1.8E+01				102.3		L	01:41			130							

STL Richland

rptFeadRadEdd v3.68

U Qual - Analyzed for, but the result is less than the Mdc or gamma scan did not identify the nuclide.  
 J Qual - No U qualifier has been assigned and the result is below the Reporting Limit (CRDL).  
 B Qual- Analyte was found in the associated laboratory blank above the MDC.

2

Monday, March 05, 2007

## STL Richland QC Duplicate Report

Lab Code: STLRL

FormNbr: R

FormatType: FEAD

VersionNbr: 05

File Name: h:\Reportdb\edd\FeadIV\Rad\W05078.Edd, h:\Reportdb\edd\FeadIV\Rad\34589.Edd

Lab Sample Id: JLPEJ1CR

Sdg/Rept Nbr: W05078

34589

Collection Date: 12/14/2006 09:23

Client Id: B1KPD6

Matrix: WATER

WATER

Sample On Date:

Moisture/Solids%\*:

QC Type: DUP

Received Date: 12/14/2006

SAF Nbr	Contract Nbr	Test User	Case Nbr	SAS Nbr	Suffix	Decant	Distilled Volume	File Id	FSuffix	RType					
S07-010	MW6-SBB-A19981								AB	H					
Batch # / Qc Type	Analyt/ CAS#	Result/ Orig Rst	Unit	Tot/Cnt Uncert 2S	Qu- al	MDC	Tracer Yield	Spk Conc/ %Rec	Analy Method	Aliq Size/	Date/Time Analyzed	RPD/ UCL	RER/ UCL	LCS LCL/UCL	R Typ
6353652	H-3	1.27E+02	pCi/L	2.5E+01		5.57E+00	100.0		TRITIUM_ELE	1.50E-01	02/28/2007	20.0	1.6		D
DUP	10028-17-8	1.55E+02		9.6E+00						L	04:16	20.0	3		

STL Richland

rptFeadRadEdd v3.68

U Qual - Analyzed for, but the result is less than the Mdc or gamma scan did not identify the nuclide.

J Qual - No U qualifier has been assigned and the result is below the Reporting Limit (CRDL).

B Qual- Analyte was found in the associated laboratory blank above the MDC.

Lot No., Due Date: J6L180167; 01/29/2007  
Client, Site: 384868; PGW 615HANFORD HANFORD  
QC Batch No., Method Test: 6353652; RH3EE H3EE by LSC  
SDG, Matrix: W05078; WATER

34589

**1.0 COC**

1.1 Is the ICOC page complete; includes all applicable analysis, dates, SOP numbers, and revisions? Yes No N/A

☒**2.0 QC Batch**

2.1 Do the Summary/Detailed Reports include a calculated result for each sample listed on the QC Batch Sheet? Yes No N/A

☒

2.2 Are the QC appropriate for the analysis included in the batch? Yes No N/A

☒

2.3 Is the Analytical Batch Worksheet complete; includes as appropriate, volumes, count times, etc? Yes No N/A

☒

2.4 Does the Worksheets include a Tracer Vial label for each sample? Yes No N/A

☒**3.0 QC & Samples**

3.1 Is the blank results, yield, and MDA within contract limits? Yes No N/A

☒

3.2 Is the LCS result, yield, and MDA within contract limits? Yes No N/A

☒

3.3 Are the MS/MSD results, yields, and MDA within contract limits? Yes No N/A

☒

3.4 Are the duplicate result, yields, and MDAs within contract limits? Yes No N/A

☒

3.5 Are the sample yields and MDAs within contract limits? Yes No N/A

☒**4.0 Raw Data**

4.1 Were results calculated in the correct units? Yes No N/A

☒

4.2 Were analysis volumes entered correctly? Yes No N/A

☒

4.3 Were Yields entered correctly? Yes No N/A

☒

4.4 Were spectra reviewed/meet contractual requirements? Yes No N/A

☒

4.5 Were raw counts reviewed for anomalies? Yes No N/A

☒**5.0 Other**

5.1 Are all nonconformances included and noted? Yes No N/A

☒

5.2 Are all required forms filled out? Yes No N/A

☒

5.3 Was the correct methodology used? Yes No N/A

☒

5.4 Was transcription checked? Yes No N/A

☒

5.5 Were all calculations checked at a minimum frequency? Yes No N/A

☒

5.6 Are worksheet entries complete and correct? Yes No N/A

☒

6.0 Comments on any No response:

First Level Review



Date

2/28/07



# STL

Data Review Checklist  
RADIOCHEMISTRY  
Second Level Review

OC Batch Number:

6 353652  
W05078

Review Item	Yes (✓)	No (✓)	N/A (✓)
A. Sample Analysis			
1. Are the sample yields within acceptance criteria?	✓		
2. Is the sample Minimum Detectable Activity < the Contract Detection Limit?	✓		
3. Are the correct isotopes reported?	✓		
B. QC Samples			
1. Is the Minimum Detectable Activity for the blank result ≤ the Contract Detection Limit?	✓		
2. Does the blank result meet the Contract criteria?	✓		
3. Is the blank result < the Contract Detection Limit?	✓		
4. Is the blank result > the Contract Detection Limit but the sample result < the Contract Detection Limit?			✓
5. Is the LCS recovery with contract acceptance criteria?	✓		
7. Is the LCS Minimum Detectable Activity ≤ the Contract Detection Limit?	✓		
8. Do the MS/MSD results and yields meet acceptance criteria?			✓
9. Do the duplicate sample results and yields meet acceptance criteria?	✓		
C. Other			
1. Are all Nonconformances included and noted?			✓
2. Are all required forms filled out?	✓		
3. Was the correct methodology used?	✓		
4. Was transcription checked?	✓		
5. Were all calculations checked at a minimum frequency?	✓		
6. Were units checked?	✓		

Comments on any "No" response:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Second Level Review:

*Sheryl A. Adam*

Date: 2-28-07

80442



# STL

## Sample Check-in List

Date/Time Received: 12-14-06 1330

Client: P&W SDG #: W05078 NA ☐ SAF #: 807-010 NA ☐

Work Order Number: UGL180167 Chain of Custody #: 807-010-2

Shipping Container ID: SAW5115 Air Bill #: N/A

1. Custody Seals on shipping container intact? NA ☐ Yes ☒ No ☐
2. Custody Seals dated and signed? NA ☐ Yes ☒ No ☐
3. Chain of Custody record present? Yes ☒ No ☐
4. Cooler temperature: \_\_\_\_\_ NA ☒ 5. Vermiculite/packing materials is NA ☒ Wet ☐ Dry ☐
6. Number of samples in shipping container: 1
7. Sample holding times exceeded? NA ☒ Yes ☐ No ☐
8. Samples have:  
\_\_\_\_ tape \_\_\_\_\_ hazard labels  
\_\_\_\_ custody seals \_\_\_\_\_ appropriate samples labels
9. Samples are:  
\_\_\_\_ in good condition \_\_\_\_\_ leaking  
\_\_\_\_ broken \_\_\_\_\_ have air bubbles  
(Only for samples requiring head space)
10. Sample pH taken? NA ☐ pH<2 ☐ pH>2 ☒ pH>9 ☐
11. Sample Location, Sample Collector Listed? \* Yes ☒ No ☐  
\*For documentation only. No corrective action needed.
12. Were any anomalies identified in sample receipt? Yes ☐ No ☒
13. Description of anomalies (include sample numbers): N/A

Sample Custodian: S. Smith Date: 12-14-06 1330

Client Sample ID	Analysis Requested	Condition	Comments/Action

Client Informed on \_\_\_\_\_ by \_\_\_\_\_ Person contacted \_\_\_\_\_

[ ] No action necessary; process as is.

Project Manager \_\_\_\_\_ Date \_\_\_\_\_

LS-023, 9/03, Rev. 5

12/19/2006 3:58:50 PM

## Sample Preparation/Analysis

Balance Id: 12924

384868, Pacific Northwest National Laboratory ,  
Pacific Northwest National LabAS H-3 Prp/SepRC5024  
U3 Enriched Tritium by Liquid Scint  
51 CLIENT: HANFORD

Pipet #:

AnalyDueDate: 01/29/2007 W05078

Sep1 DT/Tm Tech: 2-13-07

Batch: 6353652 WATER pCi/L

PM, Quote: SA , 57671

Sep2 DT/Tm Tech:

SEQ Batch, Test: None

Prep Tech:

Work Order, Lot, Sample Date/Time	Total Amt/Unit	Initial Aliquot Amt/Unit	QC Tracer Prep Date	Count Time Min	Detector Id	Count On   Off (24hr) Circle	CR Analyst, Init/Date	Comments:
1 JLPEJ-1-AA J6L180167-1-SAMP 12/14/2006 09:23 AmtRec: 20ML,3XLP #Containers: 4 Scr: Alpha: Beta:								
2 JLPEJ-1-AC-X J6L180167-1-DUP 12/14/2006 09:23 AmtRec: 20ML,3XLP #Containers: 4 Scr: Alpha: Beta:								
3 JLTJW-1-AA-B J6L190000-652-BLK 12/14/2006 09:23 AmtRec: #Containers: 1 Scr: Alpha: Beta:								
4 JLTJW-1-AC-C J6L190000-652-LCS 12/14/2006 09:23 AmtRec: #Containers: 1 Scr: Alpha: Beta:								
5 JLTJW-1-AD-BN J6L190000-652-IBLK 12/14/2006 09:23 AmtRec: #Containers: 1 Scr: Alpha: Beta:								
Comments:								
All Clients for Batch: 384868, Pacific Northwest National Laboratory Pacific Northwest National Lab, SA , 57671								
JLPEJ1AA-SAMP Constituent List: H-3 RDL:1.00E+01 pCi/L LCL:70 UCL:130 RPD:20								
STL Richland Key: In - Initial Amt, fi - Final Amt, di - Diluted Amt, s1 - Sep1, s2 - Sep2 Page 1 ISV - Insufficient Volume for Analysis WO Cnt: 5 Richland Wa. pd - Prep Dt, r - Reference Dt, ec-Enrichment Cell, ct-Cocktailed Added ICOC v4.8.26								

2/28/2007 9:36:38 AM

## ICOC Fraction Transfer/Status Report

ByDate: 2/28/2006, 3/5/2007, Batch: '6353652', User: \*ALL Order By DateTimeAccepting

Q Batch	Work Ord	CurStatus	Accepting	Comments
<b>6353652</b>				
AC	CalcC	McDowellID	2/6/2007 10:33:01	
SC		wagarr	IsBatched	12/19/2006 4:02:34 PM
SC		ICOC	IsRpt	1/30/2007 4:31:06 AM
SC		McDowellID	InSep1	2/6/2007 10:33:01 AM
SC		McDowellID	Sep1C	2/27/2007 3:28:49 PM
SC		DAWKINSO	InCnt1	2/27/2007 4:03:13 PM
SC		BlackCL	CalcC	2/28/2007 6:27:54 AM
AC		McDowellID	2/27/2007 3:28:49 PM	ICOC_RADCALC v4.8.26
AC		DAWKINSO	2/27/2007 4:03:13 PM	ICOC_RADCALC v4.8.17
AC		BlackCL	2/28/2007 6:27:54	RICH-RC-5024 REVISION 2
				RICH-RC-5024 REVISION 2
				RICH-RD-0001 REVISION 3
				RICH-RD-0001 REVISION 3

AC: Accepting Entry; SC: Status Change

STL Richland

Richland Wa.